```
11:Atom  12:Atom  13:Atom  14:Atom  15:CLASS  16:CLASS  17:CLASS  18:CLASS
19:CLASS  20:CLASS  21:CLASS
22:CLASS
```

**********INVENTOR RESULTS**********

```
=> d que l9
L3          104 SEA FILE=HCAPLUS ABB=ON  PLU=ON  ("HOLMES I"/AU OR "HOLMES I
                B"/AU OR "HOLMES I F"/AU OR "HOLMES I H"/AU OR "HOLMES I P"/AU
                OR "HOLMES IAN"/AU OR "HOLMES IAN B"/AU OR "HOLMES IAN D"/AU
                OR "HOLMES IAN F"/AU OR "HOLMES IAN H"/AU OR "HOLMES IAN
                HAMILTON"/AU OR "HOLMES IAN P"/AU OR "HOLMES IAN PETER"/AU)
L4           99 SEA FILE=HCAPLUS ABB=ON  PLU=ON  ("WATSON S"/AU OR "WATSON S
                P"/AU)
L5          164 SEA FILE=HCAPLUS ABB=ON  PLU=ON  ("WATSON STEFAN"/AU OR
                "WATSON STEPHEN"/AU OR "WATSON STEPHEN PAUL"/AU OR "WATSON
                STEPHEN PAUL"/AU OR "WATSON STEVE"/AU OR "WATSON STEVE P"/AU
                OR "WATSON STEVEN"/AU OR "WATSON STEVEN P"/AU)
L6          263 SEA FILE=HCAPLUS ABB=ON  PLU=ON  (L4 OR L5)
L7            4 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L3 AND L6
L8            6 SEA FILE=HCAPLUS ABB=ON  PLU=ON  (L3 OR L4 OR L5) AND METALLOPR
                OTEINASE?
L9            6 SEA FILE=HCAPLUS ABB=ON  PLU=ON  (L7 OR L8)


=> d que l17
L10        5752 SEA WATSON S?/AU
L11         587 SEA HOLMES I?/AU
L12           8 SEA L10 AND L11
L13         131 SEA (L10 OR L11) AND METALLOPROTEINASE?
L14          97 SEA L13 AND (METALLOPROTEINASE?(L) INHIBIT?)
L15          86 SEA L14 AND (PY<2005 OR AY<2005 OR PRY<2005)
L16          43 DUP REM L15 (43 DUPLICATES REMOVED)
L17          47 SEA (L12 OR L16)


=> dup rem l9,l17
FILE 'HCAPLUS' ENTERED AT 09:49:06 ON 26 MAR 2007
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```
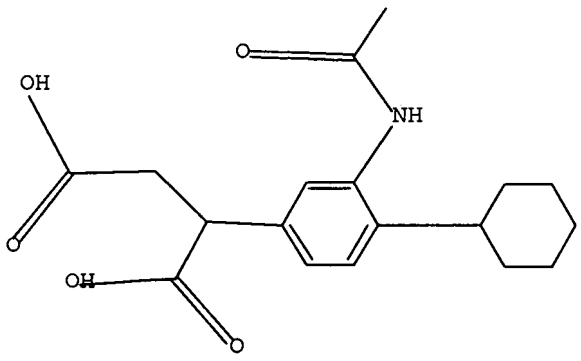
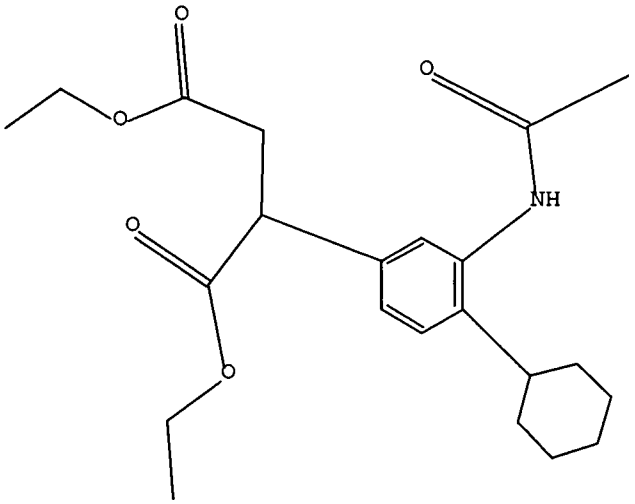

NG7 2RD, England.
LANGUAGE: English
DOCUMENT TYPE: Journal
FIELD AVAIL.: AB; LA; CT
FILE SEGMENT: Literature
AN 1994-23213 DRUGU P Full-text
AB Matrix ***metalloproteinases*** are known to play a role in the progression of human colorectal cancer. In the present study, the ***metalloproteinase inhibitor***, BB94, given by the i.p. route, ***inhibited*** experimental metastasis and ascites formation of a human colorectal tumor cell-line, C170HM2, in nude mice. Agents which ***inhibit*** the activity of invasive enzymes may reduce tumor spread and may therefore be of clinical value. (congress abstract).
ABEX C170HM2 has been selected to invade the liver following i.p. injection into nude mice. The C170HM2 tumors express both interstitial collagenase, at the leading edge of the tumor, and 72kDa gelatinase, during invasion within the liver. BB94 was administered at a dose of 40 mg/kg, i.p., from day 10 to the end of the study (day 39) and was shown to significantly reduce both the number (35% of vehicle-treated controls) and the cross-sectional area (73% of control) of the liver tumors. Histological analysis showed that the zone of proliferative cells was reduced and necrosis within the tumors was more advanced in the BB94-treated group. An ascites variant of C170HM2 has been derived in SCID mice following i.p. administration of cells. BB94 given from day 0, at the same dosage schedule as described, reduced (i) the number of mice developing ascites from 100% to 53%; (ii) the mean ascites volume from 1.78 ml to 0.38 ml; and (iii) peritoneal tumor weight from 2.19 g to 1.70 g. All the in-vivo studies were performed according to the UK coordinating committee for Cancer Research Guidelines. (NPH)

**********RESULTS FROM QUERY**********

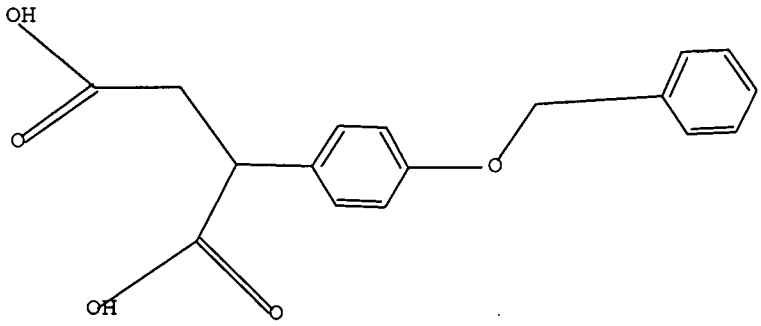
=> d que l43
L19 STR



Structure attributes must be viewed using STN Express query preparation.
L20 STR



Structure attributes must be viewed using STN Express query preparation.
L22 STR

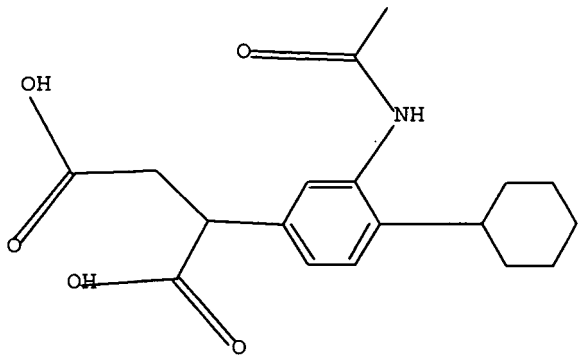


Structure attributes must be viewed using STN Express query preparation.

```
L27           1 SEA FILE=REGISTRY SSS FUL L19
L28           1 SEA FILE=REGISTRY SSS FUL L20
L30           4 SEA FILE=REGISTRY SSS FUL L22
L33           1 SEA FILE=REGISTRY ABB=ON  PLU=ON  66123-61-3
L39           1 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L27
L40           1 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L28
L41           1 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L33
L42           3 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L30
L43           4 SEA FILE=HCAPLUS ABB=ON  PLU=ON  (L39 OR L40 OR L41 OR L42)
```

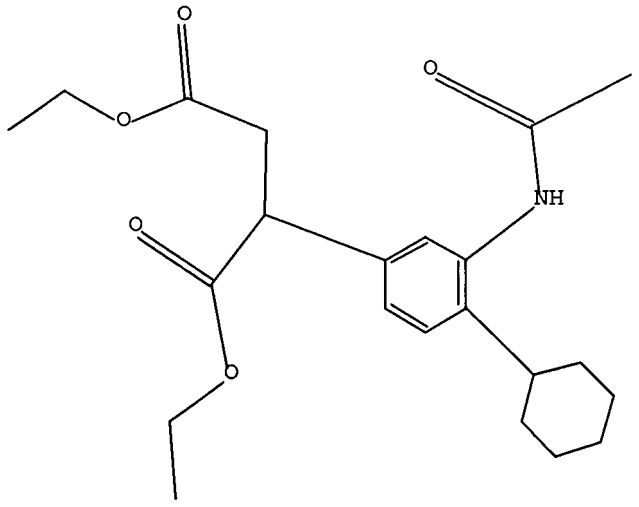
```
=> d que l68
L58        1373 SEA FILE=REGISTRY ABB=ON  PLU=ON  C18 H18 O6/MF
L59        1659 SEA FILE=REGISTRY ABB=ON  PLU=ON  C17 H16 O5/MF
L60         549 SEA FILE=REGISTRY ABB=ON  PLU=ON  C22 H31 N O5/MF
L61        1469 SEA FILE=REGISTRY ABB=ON  PLU=ON  C18 H23 N O5/MF
```

```
L62          5050 SEA FILE=REGISTRY ABB=ON  PLU=ON  (L58 OR L59 OR L60 OR L61)
L63          6773 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L62
L64         33837 SEA FILE=HCAPLUS ABB=ON  PLU=ON  METALLOPROTEINASE+NT/CT
L65         25598 SEA FILE=HCAPLUS ABB=ON  PLU=ON  METALLOPROTEINASE?
L66            47 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L63 AND (L64 OR L65)
L67            28 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L66 AND (METALLOPROTEINASE?(L)
                  INHIBIT?)
L68            24 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L67 AND (PY<2005 OR AY<2005
                  OR PRY<2005)


=> d que l90
L19               STR
```



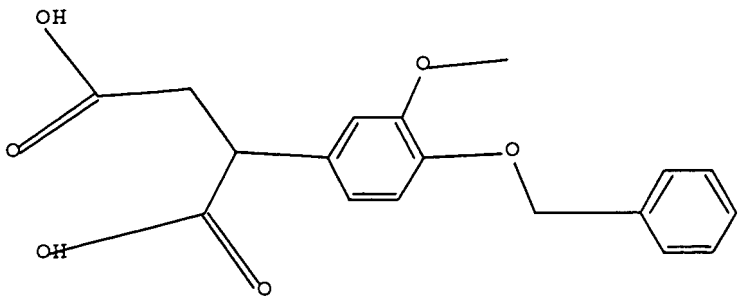
Structure attributes must be viewed using STN Express query preparation.

L20 STR

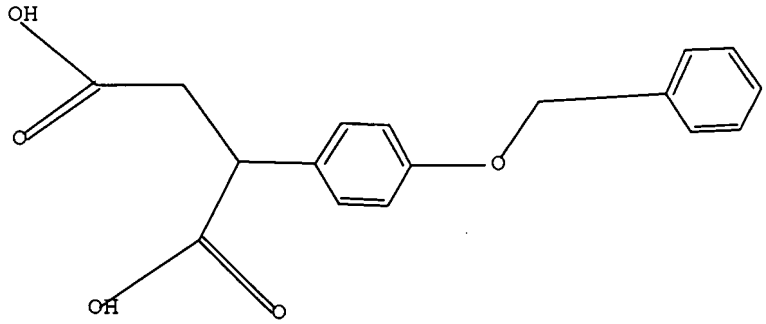


Structure attributes must be viewed using STN Express query preparation.

L21 STR



Structure attributes must be viewed using STN Express query preparation.
L22 STR



Structure attributes must be viewed using STN Express query preparation.

```
L64       33837 SEA FILE=HCAPLUS ABB=ON  PLU=ON  METALLOPROTEINASE+NT/CT
L65       25598 SEA FILE=HCAPLUS ABB=ON  PLU=ON  METALLOPROTEINASE?
L70         201 SEA FILE=MARPAT SSS FUL L19
L72         163 SEA FILE=MARPAT SSS FUL L20
L74         268 SEA FILE=MARPAT SSS FUL L21
L77         310 SEA FILE=MARPAT SSS FUL L22
L78         199 SEA FILE=MARPAT ABB=ON  PLU=ON  L70/COM
L79         161 SEA FILE=MARPAT ABB=ON  PLU=ON  L72/COM
L80         263 SEA FILE=MARPAT ABB=ON  PLU=ON  L74/COM
L81         305 SEA FILE=MARPAT ABB=ON  PLU=ON  L77/COM
L82         199 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L78
L83         161 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L79
L84         263 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L80
L85         305 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L81
L86         485 SEA FILE=HCAPLUS ABB=ON  PLU=ON  (L82 OR L83 OR L84 OR L85)
L87           7 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L86 AND (L64 OR L65)
L88           6 SEA FILE=HCAPLUS ABB=ON  PLU=ON  L87 AND (PY<2005 OR AY<2005
                OR PRY<2005)
L89           6 SEA FILE=MARPAT ABB=ON  PLU=ON  L88
L90           6 SEA FILE=MARPAT ABB=ON  PLU=ON  L89 AND (L78 OR L79 OR L80 OR
                L81)
```

=> dup rem l43,l68,l90

```
                -6/BI OR 845786-26-7/BI OR 845786-27-8/BI OR 98946-18-0/BI)
                D SCAN

     FILE 'STNGUIDE' ENTERED AT 09:38:27 ON 26 MAR 2007

     FILE 'REGISTRY' ENTERED AT 09:40:53 ON 26 MAR 2007
                E 3-ACETYLAMINO-4-CYCLOHEXYLPHENYL-BUTANEDIOIC ACID/CN
                E BUTANEDIOIC ACID/CN

     FILE 'HCAPLUS' ENTERED AT 09:43:07 ON 26 MAR 2007
                E HOLMES I/AU
L3           104 SEA ABB=ON  PLU=ON  ("HOLMES I"/AU OR "HOLMES I B"/AU OR
                "HOLMES I F"/AU OR "HOLMES I H"/AU OR "HOLMES I P"/AU OR
                "HOLMES IAN"/AU OR "HOLMES IAN B"/AU OR "HOLMES IAN D"/AU OR
                "HOLMES IAN F"/AU OR "HOLMES IAN H"/AU OR "HOLMES IAN HAMILTON"
                /AU OR "HOLMES IAN P"/AU OR "HOLMES IAN PETER"/AU)
                E WATCON S/AU
                E WATSON S/AU
L4            99 SEA ABB=ON  PLU=ON  ("WATSON S"/AU OR "WATSON S P"/AU)
                E WATSON S/AU
L5           164 SEA ABB=ON  PLU=ON  ("WATSON STEFAN"/AU OR "WATSON STEPHEN"/AU
                OR "WATSON STEPHEN PAUL"/AU OR "WATSON STEPHEN PAUL"/AU OR
                "WATSON STEVE"/AU OR "WATSON STEVE P"/AU OR "WATSON STEVEN"/AU
                OR "WATSON STEVEN P"/AU)
L6           263 SEA ABB=ON  PLU=ON  (L4 OR L5)
L7             4 SEA ABB=ON  PLU=ON  L3 AND L6
L8             6 SEA ABB=ON  PLU=ON  (L3 OR L4 OR L5) AND METALLOPROTEINASE?
L9             6 SEA ABB=ON  PLU=ON  (L7 OR L8)

     FILE 'HCAPLUS, MEDLINE, EMBASE, BIOSIS, DRUGU, WPIX' ENTERED AT 09:45:52
     ON 26 MAR 2007
L10         5752 SEA ABB=ON  PLU=ON  WATSON S?/AU
L11          587 SEA ABB=ON  PLU=ON  HOLMES I?/AU
L12            8 SEA ABB=ON  PLU=ON  L10 AND L11
L13          131 SEA ABB=ON  PLU=ON  (L10 OR L11) AND METALLOPROTEINASE?
L14           97 SEA ABB=ON  PLU=ON  L13 AND (METALLOPROTEINASE?(L) INHIBIT?)
L15           86 SEA ABB=ON  PLU=ON  L14 AND (PY<2005 OR AY<2005 OR PRY<2005)
L16           43 DUP REM L15 (43 DUPLICATES REMOVED)
                     ANSWERS '1-16' FROM FILE HCAPLUS
                     ANSWERS '17-19' FROM FILE MEDLINE
                     ANSWERS '20-31' FROM FILE BIOSIS
                     ANSWERS '32-43' FROM FILE DRUGU
L17           47 SEA ABB=ON  PLU=ON  (L12 OR L16)

     FILE 'STNGUIDE' ENTERED AT 09:48:56 ON 26 MAR 2007
                D QUE L9
                D QUE L17

     FILE 'HCAPLUS, MEDLINE, BIOSIS, DRUGU, WPIX' ENTERED AT 09:49:06 ON 26
     MAR 2007
L18           44 DUP REM L9 L17 (9 DUPLICATES REMOVED)
                     ANSWERS '1-17' FROM FILE HCAPLUS
                     ANSWERS '18-20' FROM FILE MEDLINE
                     ANSWERS '21-32' FROM FILE BIOSIS
                     ANSWERS '33-44' FROM FILE DRUGU
                D IBIB ABS HITSTR RETABLE L18 1-17
                D IBIB ABS L18 18-44

     FILE 'REGISTRY' ENTERED AT 10:03:00 ON 26 MAR 2007
                E BUTANEDIOIC ACID/CNS
```

```
                E ACETYLAMINO/CNS AND CYCLOHEXYLPHENYL/CNS

     FILE 'STNGUIDE' ENTERED AT 10:06:18 ON 26 MAR 2007

     FILE 'REGISTRY' ENTERED AT 10:13:17 ON 26 MAR 2007
L19             STRUCTURE UPLOADED
L20             STRUCTURE UPLOADED
L21             STRUCTURE UPLOADED
L22             STRUCTURE UPLOADED
L23           0 SEA SSS SAM L19
L24           0 SEA SSS SAM L20
L25           0 SEA SSS SAM L21
L26           0 SEA SSS SAM L22
L27           1 SEA SSS FUL L19
                D SCAN
L28           1 SEA SSS FUL L20
                D SCAN
L29           2 SEA SSS FUL L21
                D SCAN
L30           4 SEA SSS FUL L22
                D SCAN
                D SCAN L27
                D BROW L27
L31           0 SEA ABB=ON  PLU=ON  38913-13-2/CRN
                D BROW L28
L32           0 SEA ABB=ON  PLU=ON  38913-20-1/CRN
                D BROW L29
L33           1 SEA ABB=ON  PLU=ON  66123-61-3
L34           0 SEA ABB=ON  PLU=ON  66123-61-3/CRN
                D BROW L30
                D RN L30 1-4
L35           0 SEA ABB=ON  PLU=ON  117726-66-6/CRN
L36           0 SEA ABB=ON  PLU=ON  103271-91-6/CRN
L37           0 SEA ABB=ON  PLU=ON  66123-61-3/CRN
L38           0 SEA ABB=ON  PLU=ON  66123-34-0/CRN
                D SCAN L27
                E BUTANEDIOIC ACID, [3-(ACETYLAMINO)-4-CYCLOHEXYLPHENYL]-/CN
                D BROW L27
                E BUTANEDIOIC ACID, [3-(ACETYLAMINO)-4-CYCLOHEXYLPHENYL]/CN

     FILE 'HCAPLUS' ENTERED AT 10:21:02 ON 26 MAR 2007
L39           1 SEA ABB=ON  PLU=ON  L27
L40           1 SEA ABB=ON  PLU=ON  L28
L41           1 SEA ABB=ON  PLU=ON  L33
L42           3 SEA ABB=ON  PLU=ON  L30
L43           4 SEA ABB=ON  PLU=ON  (L39 OR L40 OR L41 OR L42)
                D BIB TOT

     FILE 'REGISTRY' ENTERED AT 10:21:42 ON 26 MAR 2007
L44           6 SEA ABB=ON  PLU=ON  (L27 OR L28 OR L33 OR L30)
                D BROW

     FILE 'HCAPLUS' ENTERED AT 10:22:32 ON 26 MAR 2007
L45           0 SEA ABB=ON  PLU=ON  L43 AND METALLOPROTEINASE?

     FILE 'BEILSTEIN' ENTERED AT 10:23:16 ON 26 MAR 2007
L46           1 SEA SSS FUL L19
L47           1 SEA SSS FUL L20
L48           0 SEA SSS FUL L21
L49           1 SEA SSS FUL L22
```

```
L50             0 SEA ABB=ON  PLU=ON  L46 NOT L27
L51             0 SEA ABB=ON  PLU=ON  L47 NOT L28
L52             0 SEA ABB=ON  PLU=ON  L49 NOT L30

     FILE 'REGISTRY' ENTERED AT 10:24:52 ON 26 MAR 2007
                D BROW L33
                D BROW L30
L53             1 SEA ABB=ON  PLU=ON  66123-34-0
L54             0 SEA ABB=ON  PLU=ON  66123-34-0/CRN

     FILE 'HCAPLUS' ENTERED AT 10:26:44 ON 26 MAR 2007
L55             1 SEA ABB=ON  PLU=ON  L53
L56             4 SEA ABB=ON  PLU=ON  (L43 OR L55)

     FILE 'REGISTRY' ENTERED AT 10:26:58 ON 26 MAR 2007
L57             4 SEA ABB=ON  PLU=ON  (L27 OR L28 OR L33 OR L53)
                D BROW
L58          1373 SEA ABB=ON  PLU=ON  C18 H18 O6/MF
L59          1659 SEA ABB=ON  PLU=ON  C17 H16 O5/MF
L60           549 SEA ABB=ON  PLU=ON  C22 H31 N O5/MF
L61          1469 SEA ABB=ON  PLU=ON  C18 H23 N O5/MF
L62          5050 SEA ABB=ON  PLU=ON  (L58 OR L59 OR L60 OR L61)

     FILE 'HCAPLUS' ENTERED AT 10:28:38 ON 26 MAR 2007
L63          6773 SEA ABB=ON  PLU=ON  L62
                E METALLOPROTEINASE/CT
                E E3+ALL
L64         33837 SEA ABB=ON  PLU=ON  METALLOPROTEINASE+NT/CT
                E METALLOPROTEINASE/CT
L65         25598 SEA ABB=ON  PLU=ON  METALLOPROTEINASE?
L66            47 SEA ABB=ON  PLU=ON  L63 AND (L64 OR L65)
L67            28 SEA ABB=ON  PLU=ON  L66 AND (METALLOPROTEINASE?(L)INHIBIT?)
                D KWIC
                D KWIC 2
L68            24 SEA ABB=ON  PLU=ON  L67 AND (PY<2005 OR AY<2005 OR PRY<2005)

     FILE 'MARPAT' ENTERED AT 10:30:35 ON 26 MAR 2007
L69             5 SEA SSS SAM L19
L70           201 SEA SSS FUL L19
L71             4 SEA SSS SAM L20
L72           163 SEA SSS FUL L20
L73             6 SEA SSS SAM L21
L74           268 SEA SSS FUL L21
L75             6 SEA SSS SAM L22
L76             6 SEA SSS SAM L22
L77           310 SEA SSS FUL L22
L78           199 SEA ABB=ON  PLU=ON  L70/COM
L79           161 SEA ABB=ON  PLU=ON  L72/COM
L80           263 SEA ABB=ON  PLU=ON  L74/COM
L81           305 SEA ABB=ON  PLU=ON  L77/COM

     FILE 'HCAPLUS' ENTERED AT 10:34:49 ON 26 MAR 2007
L82           199 SEA ABB=ON  PLU=ON  L78
L83           161 SEA ABB=ON  PLU=ON  L79
L84           263 SEA ABB=ON  PLU=ON  L80
L85           305 SEA ABB=ON  PLU=ON  L81
L86           485 SEA ABB=ON  PLU=ON  (L82 OR L83 OR L84 OR L85)
L87             7 SEA ABB=ON  PLU=ON  L86 AND (L64 OR L65)
L88             6 SEA ABB=ON  PLU=ON  L87 AND (PY<2005 OR AY<2005 OR PRY<2005)
```

```
     FILE 'HCAPLUS' ENTERED AT 10:35:36 ON 26 MAR 2007
L*** DEL       6 S L88

     FILE 'MARPAT' ENTERED AT 10:35:52 ON 26 MAR 2007
L89            6 SEA ABB=ON  PLU=ON  L88
L90            6 SEA ABB=ON  PLU=ON  L89 AND (L78 OR L79 OR L80 OR L81)

     FILE 'STNGUIDE' ENTERED AT 10:36:44 ON 26 MAR 2007
                D QUE L43
                D QUE L68
                D QUE L90

     FILE 'HCAPLUS, MARPAT' ENTERED AT 10:37:05 ON 26 MAR 2007
L91           33 DUP REM L43 L68 L90 (1 DUPLICATE REMOVED)
                     ANSWERS '1-28' FROM FILE HCAPLUS
                     ANSWERS '29-33' FROM FILE MARPAT
                D IBIB ABS HITSTR RETABLE L91 1-28
                D IBIB ABS QHIT L91 29-33


     FILE HOME

     FILE HCAPLUS

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     FILE LAST UPDATED: 25 Mar 2007  (20070325/ED)

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      This file contains CAS Registry Numbers for easy and accurate
      substance identification.

     FILE REGISTRY
     Property values tagged with IC are from the ZIC/VINITI data file
     provided by InfoChem.

     STRUCTURE FILE UPDATES:   25 MAR 2007  HIGHEST RN 928121-90-8
     DICTIONARY FILE UPDATES:  25 MAR 2007  HIGHEST RN 928121-90-8

     New CAS Information Use Policies, enter HELP USAGETERMS for details.

     TSCA INFORMATION NOW CURRENT THROUGH December 2, 2006

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     predicted properties as well as tags indicating availability of
     experimental property data in the original document.  For information
     on property searching in REGISTRY, refer to:
```

http://www.cas.org/ONLINE/UG/regprops.html

FILE STNGUIDE
FILE CONTAINS CURRENT INFORMATION.
LAST RELOADED: Mar 23, 2007 (20070323/UP).

FILE MEDLINE
FILE LAST UPDATED: 24 Mar 2007 (20070324/UP). FILE COVERS 1950 TO DATE.

SDI results from March 16, 17, and 20, may have been incomplete. SDIs delivered on March 24 will include any missing records. If you have questions, please contact your STN Service Center.

All regular MEDLINE updates from November 15 to December 16 have been added to MEDLINE, along with 2007 Medical Subject Headings (MeSH(R)) and 2007 tree numbers.

The annual reload will be available in early 2007.

This file contains CAS Registry Numbers for easy and accurate substance identification.

FILE EMBASE
FILE COVERS 1974 TO 23 Mar 2007 (20070323/ED)

EMBASE is now updated daily. SDI frequency remains weekly (default) and biweekly.

This file contains CAS Registry Numbers for easy and accurate substance identification.

FILE BIOSIS
FILE COVERS 1969 TO DATE.
CAS REGISTRY NUMBERS AND CHEMICAL NAMES (CNs) PRESENT
FROM JANUARY 1969 TO DATE.

RECORDS LAST ADDED: 21 March 2007 (20070321/ED)

FILE DRUGU
FILE LAST UPDATED: 23 MAR 2007 <20070323/UP>
>>> DERWENT DRUG FILE (SUBSCRIBER) <<<

>>> FILE COVERS 1983 TO DATE <<<
>>> THESAURUS AVAILABLE IN /CT <<<

FILE WPIX
FILE LAST UPDATED: 22 MAR 2007 <20070322/UP>
MOST RECENT THOMSON SCIENTIFIC UPDATE: 200720 <200720/DW>
DERWENT WORLD PATENTS INDEX SUBSCRIBER FILE, COVERS 1963 TO DATE

>>> New reloaded DWPI Learn File (LWPI) available as well <<<

>>> YOU ARE IN THE NEW AND ENHANCED DERWENT WORLD PATENTS INDEX <<<

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>>> IPC Reform backfile reclassification has been loaded to 31 December 2006. No update date (UP) has been created for the reclassified documents, but they can be identified by 20060101/UPIC and 20061231/UPIC. <<<

FOR A COPY OF THE DERWENT WORLD PATENTS INDEX STN USER GUIDE, PLEASE VISIT:
http://www.stn-international.de/training_center/patents/stn_guide.pdf

FOR DETAILS OF THE PATENTS COVERED IN CURRENT UPDATES, SEE
http://scientific.thomson.com/support/patents/coverage/latestupdates/

PLEASE BE AWARE OF THE NEW IPC REFORM IN 2006, SEE
http://www.stn-international.de/stndatabases/details/ipc_reform.html and
http://scientific.thomson.com/media/scpdf/ipcrdwpi.pdf

>>> FOR DETAILS ON THE NEW AND ENHANCED DERWENT WORLD PATENTS INDEX PLEASE SEE
http://www.stn-international.de/stndatabases/details/dwpi_r.html <<<

FILE BEILSTEIN
FILE LAST UPDATED ON JANUARY 10, 2007

FILE COVERS 1771 TO 2006.
FILE CONTAINS 9,780,003 SUBSTANCES

>>>PLEASE NOTE: Reaction Data and substance data are stored in separate documents and can not be searched together in one query. Reaction data for BEILSTEIN compounds may be displayed immediately with the display codes PRE (preparations) and REA (reactions). A substance answer set retrieved after the search for a chemical name, a compounds with available reaction information by combining with PRE/FA, REA/FA or more generally with RX/FA. The BEILSTEIN Registry Number (BRN) is the link between a BEILSTEIN compound and belonging reactions. For mo detailed reaction searches BRNs can be searched as reaction partner BRNs Reactant BRN (RX.RBRN) or Product BRN (RX.PBRN).<<<

>>> FOR SEARCHING PREPARATIONS SEE HELP PRE <<<

****
* PLEASE NOTE THAT THERE ARE NO FORMATS FREE OF COST. *
* SET NOTICE FEATURE: THE COST ESTIMATES CALCULATED FOR SET NOTICE *
* ARE BASED ON THE HIGHEST PRICE CATEGORY. THEREFORE; THESE *
* ESTIMATES MAY NOT REFLECT THE ACTUAL COSTS. *
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NEW
** PATENT NUMBERS (PN) AND BABS ACCESSION NUMBERS (BABSAN) CAN NOW BE SEARCHED, SELECTED AND TRANSFERRED.*
** NEW DISPLAY FORMATS ALLREF, ALLP AND BABSAN SHOW ALL REFERENCES, ALL PATENT REFERENCES, OR ALL BABS ACCESSION NUMBERS FOR A COMPOUND AT A GLANCE.*

FILE MARPAT
FILE CONTENT: 1961-PRESENT VOL 146 ISS 12 (20070325/ED)

SOME MARPAT RECORDS ARE DERIVED FROM INPI DATA FOR 1961-1987

MOST RECENT CITATIONS FOR PATENTS FROM MAJOR ISSUING AGENCIES
(COVERAGE TO THESE DATES IS NOT COMPLETE):

US	2007032719	08 FEB 2007
DE	102006011317	15 FEB 2007
EP	1750119	07 FEB 2007
JP	2007035357	08 FEB 2007
WO	2007022718	01 MAR 2007
GB	2428675	07 FEB 2007
FR	2889524	09 FEB 2007
RU	2293086	10 FEB 2007
CA	2552059	19 JAN 2007

Expanded G-group definition display now available.

(reactions). A substance answer set retrieved after the search for a chemical name, a compounds with available reaction information by combining with PRE/FA, REA/FA or more generally with RX/FA. The BEILSTEIN Registry Number (BRN) is the link between a BEILSTEIN compound and belonging reactions. For mo detailed reaction searches BRNs can be searched as reaction partner BRNs Reactant BRN (RX.RBRN) or Product BRN (RX.PBRN).<<<

>>> FOR SEARCHING PREPARATIONS SEE HELP PRE <<<

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* PLEASE NOTE THAT THERE ARE NO FORMATS FREE OF COST. *
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NEW

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* *NEW DISPLAY FORMATS ALLREF, ALLP AND BABSAN SHOW ALL REFERENCES, ALL PATENT REFERENCES, OR ALL BABS ACCESSION NUMBERS FOR A COMPOUND AT A GLANCE.*

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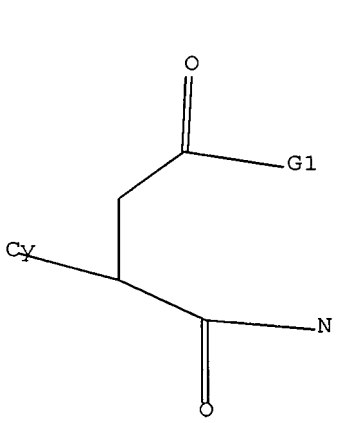
L42 STR



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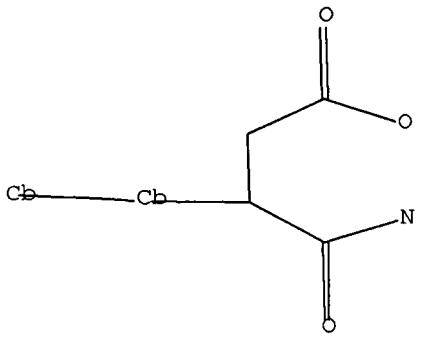

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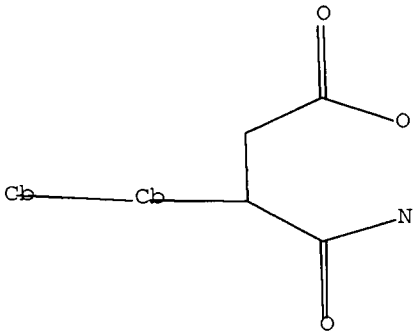

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L53 1 SEA FILE=BEILSTEIN ABB=ON PLU=ON L52 NOT L49

=> d ide allref 153 tot

L53 ANSWER 1 OF 1 BEILSTEIN COPYRIGHT 2006 BEILSTEIN MDL on STN

Beilstein Records (BRN):	3390730
Chemical Name (CN):	3-bicyclohexyl-4-yl-succinamic acid ethyl ester
Autonom Name (AUN):	3-bicyclohexyl-4-yl-succinamic acid ethyl ester
Molec. Formula (MF):	C18 H31 N O3
Molecular Weight (MW):	309.45
Lawson Number (LN):	11110, 298
Compound Type (CTYPE):	isocyclic
Constitution ID (CONSID):	3040392
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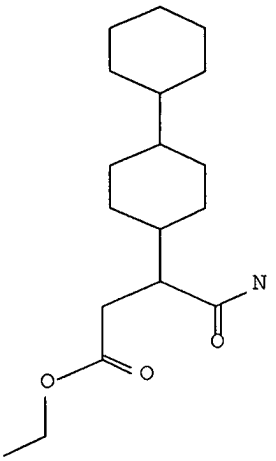
Beilstein Citation (BSO):	3-09-00-04036

Entry Date (DED): 1990/02/15
Update Date (DUPD): 1992/06/02



Field Availability:

Code	Name	Occurrence
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AUN	Autonomname	1
MF	Molecular Formula	1
FW	Formular Weight	1
LN	Lawson Number	2
CTYPE	Compound Type	1
CONSID	Constitution ID	1
TAUTID	Tautomer ID	1
BSO	Beilstein Citation	1
DED	Entry Date	1
DUPD	Update Date	1
MP	Melting Point	1

This substance also occurs in Reaction Documents:

Code	Name	Occurrence
RX	Reaction Documents	1
RXPRO	Substance is Reaction Product	1

All References:
ALLREF

1. Fieser et al., J.Amer.Chem.Soc., CODEN: JACSAT, 70, <1948>, 3177

=> file marpat
FILE 'MARPAT' ENTERED AT 15:27:43 ON 24 OCT 2006

Aliphatic Polyimides from Phenylene Bis(Succinic Anhydride) and Bis(glutaric anhydride)

TAKUMA TESHIROGI, *Macromolecular Research Laboratory, Yamagata University, Yonezawa, Yamagata, 992, Japan*

Synopsis

Meta and *para* derivatives of phenylene bis(succinic anhydride) and bis(glutaric anhydride) were obtained from 1,3- and 1,4-bis(β-cyano-β-carbethoxyvinyl)benzene with potassium cyanide or Meldrum acid followed by hydrolysis with concentrated hydrochloric acid and dehydration with acetic anhydride. Aliphatic polyimides were prepared from these anhydrides with six aromatic diamines through thermal ring closure of polyamic acids obtained by solution polymerization in dimethylacetamide, and thermal stability of these polyimides was examined by thermogravimetric analysis.

INTRODUCTION

The investigation of aliphatic polyimide derived from aliphatic tetracarboxylic dianhydride is far less common than that of its aromatic counterpart. Aliphatic tetracarboxylic dianhydrides so far used were cyclobutane,[1] butane,[2,3] neopentane,[4] cycloocta-1,5-diene,[5] and bicyclooct-2-ene tetracarboxylic dianhydrides.[6] This paper describes the preparation of phenylene bis(succinic anhydride)s, bis(glutaric anhydride)s, and novel aliphatic polyimides derived from the anhydrides.

EXPERIMENTAL

Material

1,4-Bis(β-cyano-β-carbethoxyvinyl)benzene (pBCCB)

*p*BCCB was prepared from commercially available terephthalaldehyde with ethyl cyanoacetate according to the method of Kauffmann[7] and recrystallized from dimethylacetamide (DMAc). Yield, 98%, mp 211–212°C (lit. 201°C)

1,3-Bis(β-cyano-β-carbethoxyvinyl)benzene (mBCCB)

*m*BCCB was prepared from isophthalaldehyde[8] with ethyl cyanoacetate in a manner similar to that above and recrystallized from ethanol. Yield, 77.2%, mp 141–142°C

ANAL. Calcd for $C_{18}H_{16}N_2O_4$: C, 66.66%; H, 4.97%; N, 8.64%. Found: C, 66.96%; H, 4.98%; N, 8.90%.

Journal of Polymer Science: Part A: Polymer Chemistry, Vol. 25, 31–36 (1987)
 CCC 0360-3676/87/010031-06$04.00

p-*Phenylene Bis(succinic acid)* (p*PBS*)

Into a suspension of 64.8 g (0.2 mol) of finely powdered *p*BCCB in 800 mL of ethanol was added dropwise a solution of 42 g (0.65 mol) of potassium cyanide in 200 mL of water, and the mixture was magnetically stirred at room temperature for 12 h. A clear solution was obtained. After dilution with 1 L of water, the solution was acidified to pH 3 with concentrated hydrochloric acid. The oil that precipitated was stirred until solidification and collected. The product was refluxed with 200 mL of concentrated hydrochloric acid. On chilling in a refrigerator, the solid crystallized was filtered off and recrystallized from a mixture (9/1) of water and acetic acid. Yield, 51.5%, mp 232–233°C.

ANAL. Calcd for $C_{14}H_{14}O_8$: C, 54.20%; H, 4.55%. Found C, 54.03%; H, 4.45%.

p-*Phenylene Bis(glutaric acid)* (p*PBG*)

Into a suspension of 64.8 g (0.2 mol) of finely powdered *p*BCCB in 800 mL of ethanol was added dropwise a solution of 63.4 g (0.48 mol) of Meldrum acid[9] and 20 g of sodium hydroxide in 200 mL of water and the reaction mixture was magnetically stirred at room temperature for 12 h. A clear solution obtained was worked up as described for *p*PBS. Yield, 77.1%, mp 260–261°C

ANAL. Calcd for $C_{16}H_{18}O_8$: C, 56.80%; H, 5.36%. Found: C, 56.22%; H, 5.46%.

p-*Phenylene Bis(succinic anhydride)* (p*PBSA*)

A dispersion of 12.4 g (0.04 mol) of *p*PBS in 40 mL of acetic anhydride was refluxed for 2 h. After cooling to room temperature, 40 mL of diethyl ether was added to the reaction mixture. The precipitate obtained was collected and recrystallized from acetic anhydride-diethyl ether. Yield, 85.8%, mp 195–196°C.

ANAL. Calcd for $C_{14}H_{10}O_6$: C, 61.32%; H, 3.68%. Found: C, 61.10%; H, 3.49%.

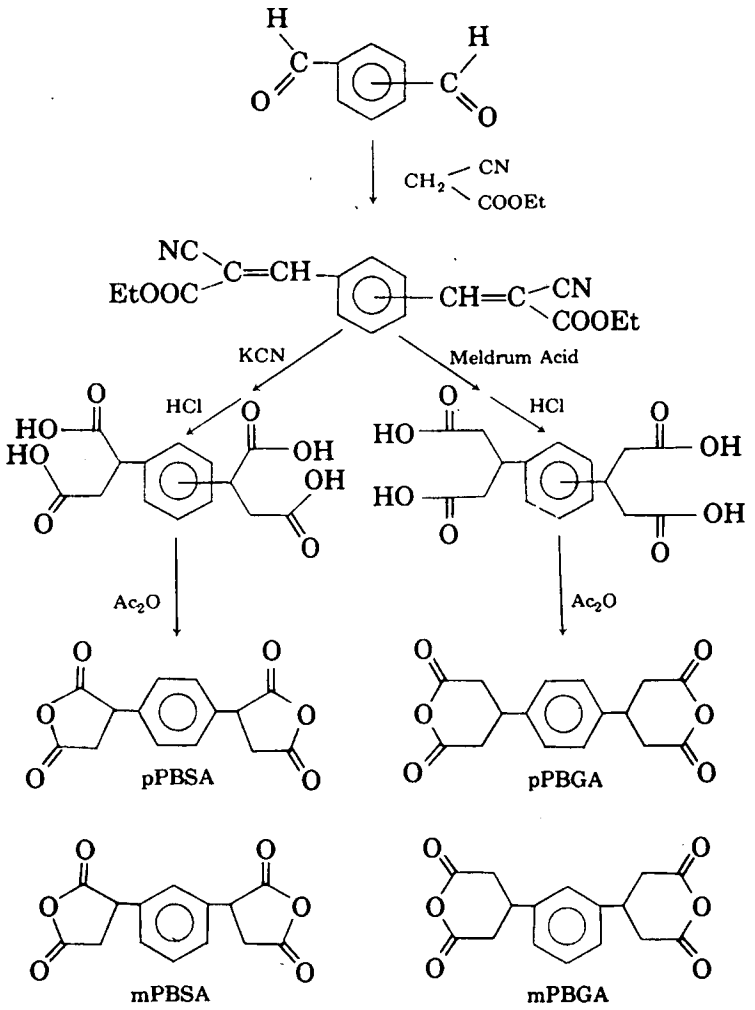
Polymerization of p*PBSA with 4,4′-Diaminodiphenyl Ether*

Into a solution of 0.4005 g (0.002 mol) of 4,4′-diamino-diphenyl ether (ODA) in 5 mL of DMAc was added, by portions, 0.5485 g (0.002 mol) of *p*PBSA. The reaction mixture was magnetically stirred at 23°C for 24 h and cast on a glass plate. The polymer film was dried at 100°C and cured at 220°C under reduced pressure.

ANAL. Calcd for $C_{26}H_{18}N_2O_5$: C, 71.23%; H, 4.14%; N, 6.39%. Found: C, 70.59%; H, 4.13%; N, 6.67%.

RESULTS AND DISCUSSION

The method for the syntheses of four aliphatic tetracarboxylic anhydrides, *m*- and *p*-phenylene bis(succinic anhydride) (*m*PBSA and *p*PBSA) and *m*- and *p*-phenylene bis(glutaric anhydride) (*m*PBGA and *p*PBGA), are shown in Scheme 1. Yields and melting points of tetracarboxylic acids and dianhydride are summarized in Table I.

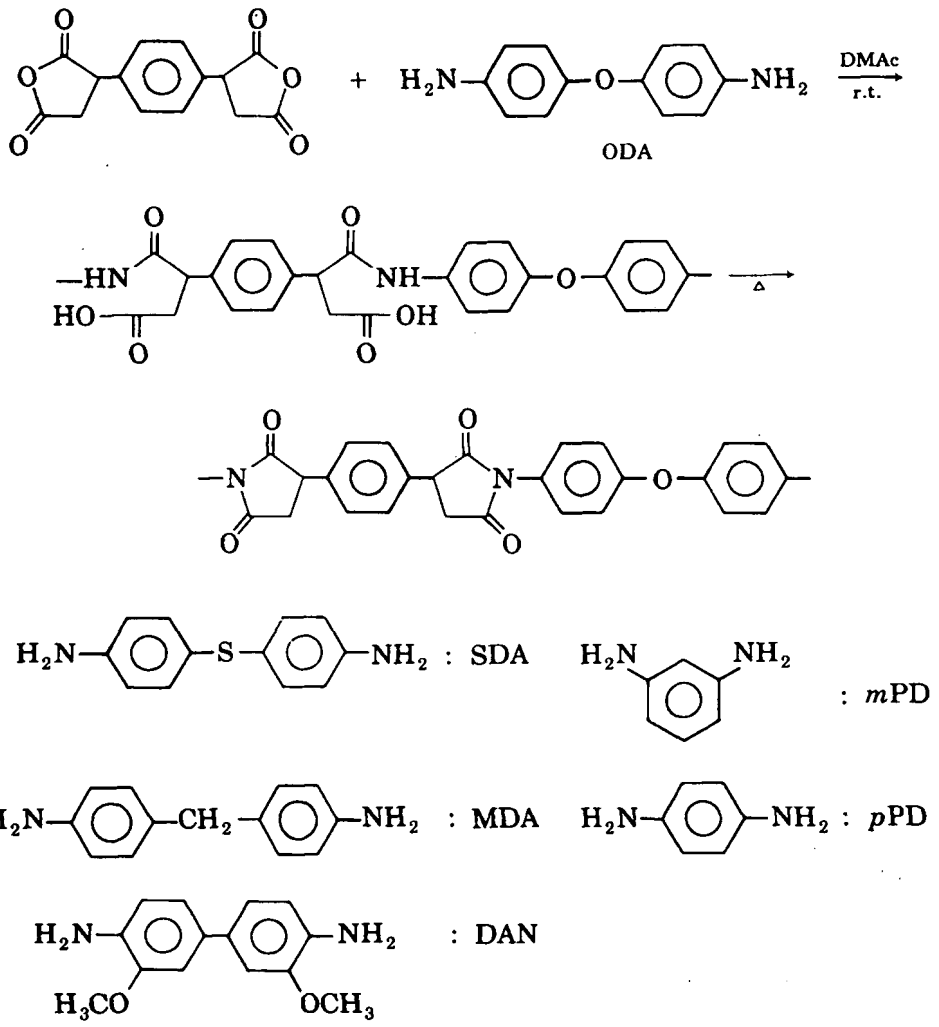


Scheme 1.

Polymerization sequence of *p*PBSA and ODA and other aromatic diamines used for polymerization are pictured in Scheme 2. IR spectra of polyamic acid and polyimide films prepared from *p*PBSA with ODA are shown in Figures 1 and 2, respectively. The intensity of several broad bands at 3300–2200 cm^{-1} assigned to carboxyl and amide groups in the IR spectrum of the polyamic acid remarkably decreased in the spectrum of polyimide, and a five-membered

TABLE I
Yield and Melting Point of Tetracarboxylic Acids and Dianhydrides

	Tetracarboxylic acid				Tetracarboxylic dianhydride			
	*p*PBS	*p*PBG	*m*PBS	*m*PBG	*p*PBSA	*p*PBGA	*m*PBSA	*m*PBGA
Yield (%)	51.0	77.1	97.3	95.0	85.8	64.9	53.3	56.3
mp (°C)	232–233	260–261	230–231	185–186	195–196	279–280	197–198	173–174



Scheme 2.

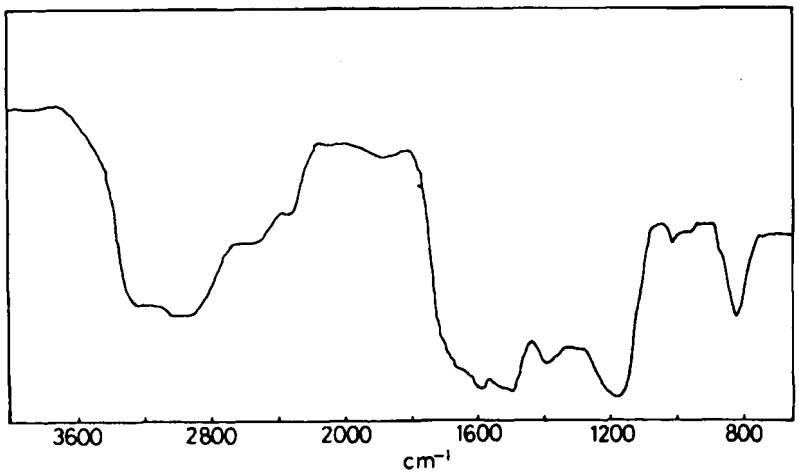


Fig. 1. IR spectrum of polyamic acid from *p*PBSA with ODA.

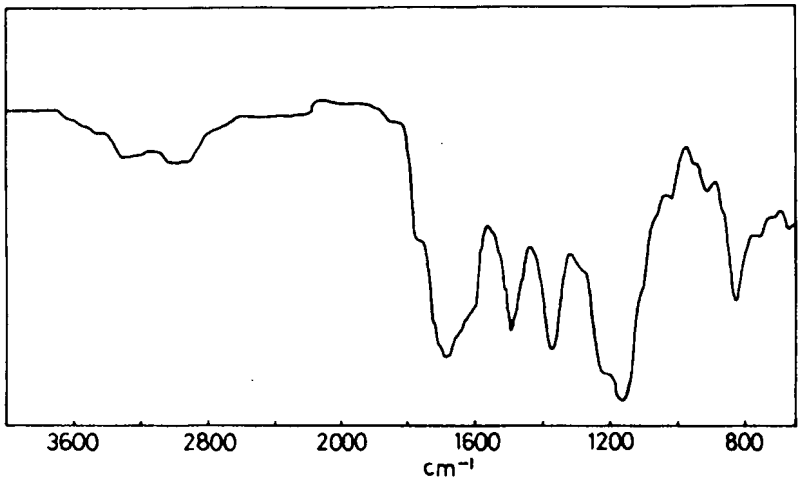


Fig. 2. IR spectrum of polyimide from *p*PBSA with ODA.

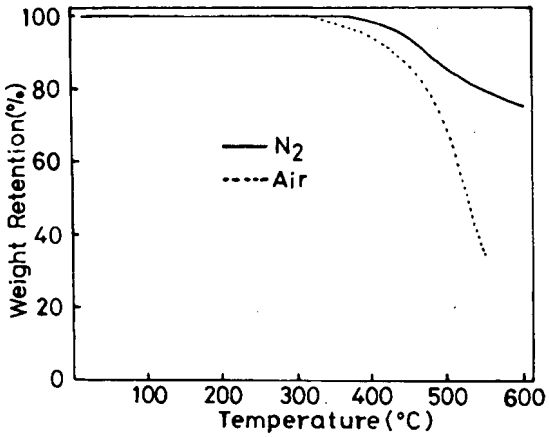


Fig. 3. Thermogravimetric analysis of polyimide film from *p*PBSA with ODA. Temperature was programmed at 5°C/min

TABLE II
Reduced Viscosity of Polyamic Acid and Thermal Stability of Polyimide

	*p*PBSA			*p*PBGA			*m*PBSA			*m*PBGA		
	$\eta^{a}_{sp/c}$	T^{b}_{10} (°C)		$\eta_{sp/c}$	T_{10} (°C)		$\eta_{sp/c}$	T_{10} (°C)		$\eta_{sp/c}$	T_{10} (°C)	
		N_2	Air		N_2	Air		N_2	Air		N_2	Air
ODA	1.00	435	430	0.79	440	405	0.62	465	440	0.63	420	400
SDA	0.53	430	430	0.35	400	390	0.47	460	455	0.38	430	425
MDA	0.76	430	430	0.63	440	425	0.55	460	405	0.53	440	435
*m*PD	0.53	460	425	0.53	430	400	0.23	440	430	0.33	420	400
*p*PD	0.61	455	415	0.63	425	390	0.27	450	400	0.53	400	370
DAN	0.75	415	405	0.70	390	355	0.28	430	380	0.38	400	380

[a] Measured in DMAc (c = 0.2 g/l) at 30°C.
[b] T_{10} is temperature at which 10% weight loss of polyimide is observed.

imide band clearly appeared at 1770 cm^{-1} in Fig. 2. Thermograms of polyimide film prepared from *p*PBSA with ODA are shown in Figure 3. The temperatures at which 10% weight loss of the polyimide was observed were 430°C under air and 435°C under nitrogen. The reduced viscosity of polyamic acids and the temperature at which 10% weight loss of polyimides was observed are summarized in Table II. The thermal stability of these polyimides was moderate to fairly high.

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